

Case 6934

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the matter of

U.S. National Phase Entry  
Under 35 USC 371 from  
the International Application of  
KENNEALLY, Corey James et al  
Int'l Application No. PCT/US98/23311  
Filed in the RO/US on 02 November 1998  
Entitled: A Process For Making High Purity Fatty  
Acid Lower Alkyl Esters

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents  
Box PCT  
Washington, D.C. 20231

Dear Sir:

Before computing the fees for entering the captioned International Application into the U.S. National Phase, please enter the following amendments **IN THE CLAIMS**:

- \* Please cancel Claims 1 thru 10.

Please add the following new claims:

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11. A process for making high purity fatty acid lower alkyl esters, comprising the steps of:
- (a) converting a source of fatty acids to a product mixture comprising fatty acid lower alkyl esters and by-products;
  - (b) water-washing the product mixture at an elevated temperature and an elevated pressure to remove at least a portion of the by-products from the product mixture; and
  - (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters; and

wherein at least a portion of the fatty acids have from about 20 to about 24 carbon atoms and further wherein the high purity fatty acid lower alkyl esters have an acid value of no greater than about 1.0.

12. A process according to claim 11, further comprising the step of collecting at least one fraction of high purity fatty acid lower alkyl esters during the step of fractionally distilling.

13. A process according to claim 12 wherein the high purity fatty acid lower alkyl esters have an acid value of less than about 0.5.

14. A process according to claim 11 wherein the step of fractionally distilling is performed at a temperature of from about 325°F to about 475°F and in the absence of base.
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15. A process according to claim 11 wherein the step of fractionally distilling is performed at a temperature of from about 475°F to about 530°F and in the presence of a base.

16. A process according to claim 15 wherein step (c) comprises the steps of:

- (1) fractionally distilling the water-washed product mixture to obtain a first portion of the fatty acid lower alkyl esters;
- (2) neutralizing the remaining water-washed product mixture to an acid value of no more than about 0.2; and
- (3) further fractionally distilling the neutralized water-washed product mixture to obtain a second portion of the fatty acid lower alkyl esters.

17. A process according to claim 11 wherein the source of fatty acids is an oil selected from the group consisting of hydrogenated and unhydrogenated fish oil, hydrogenated and unhydrogenated soybean oil, hydrogenated and unhydrogenated palm kernel oil, hydrogenated and unhydrogenated coconut oil, hydrogenated and unhydrogenated sunflower oil, hydrogenated and unhydrogenated safflower oil, hydrogenated and unhydrogenated corn oil, hydrogenated and unhydrogenated cottonseed oil, hydrogenated and unhydrogenated peanut oil, hydrogenated and unhydrogenated canola oil, hydrogenated and unhydrogenated high erucic acid rapeseed oil, and mixtures thereof.

18. A process according to claim 17 further comprising the step of subjecting the oil to at least one step selected from the group consisting of bleaching, deodorizing, hardening and alkali refining, before its conversion.

19. A process according to claim 11 further comprising the step of hardening the fatty acid lower alkyl ester.

20. A process according to claim 11 wherein the water-washed product mixture comprises no more than about 1000 ppm soap, no more than about 1000 ppm glycerides, and no more than about 100 ppm basic catalyst.

21. A process according to claim 12 wherein the fatty acid lower alkyl esters comprise behenic methyl ester.

22. A process according to claim 21 wherein the color of the behenic acid methyl ester is about 0.5 or less Lovibond yellow.

23. A process according to claim 11 wherein the water-washing elevated temperature is from about 70°F to about 200°F and the water-washing elevated pressure is from about 760 mm Hg to about 1000 mm Hg.

24. A process for making high purity fatty acid lower alkyl esters, comprising the steps of:

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cont.
- (a) converting a source of fatty acid to a product mixture comprising fatty acid lower alkyl esters and by-products;
  - (b) water-washing the product mixture at an elevated temperature and elevated pressure to remove at least a portion of the by-products from the product mixture; and
  - (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters; and

wherein the step of fractionally distilling is selected from the group consisting of fractionally distilling in the absence of base at a temperature of from about 325°F to about 475°F and fractionally distilling in the presence of base at a temperature of from about 475°F to about 530°F, and wherein at least a portion of the fatty acids have at least about 16 carbon atoms and further wherein the high purity fatty acid lower alkyl esters have an acid value of no greater than about 1.0.

25. A process according to claim 24 wherein the fatty acid lower alkyl esters comprise C18 methyl esters, C20 methyl esters and C22 methyl esters; and the process step (c) comprises the steps of collecting a first fraction comprising C18 methyl esters; collecting a second fraction comprising C18 methyl esters, C20 methyl esters and C22 methyl esters; and collecting a third fraction comprising C22 methyl esters.

26. A process according to claim 25 wherein the third fraction comprises from about 86% to about 95%, by weight, C22 methyl ester.

27. A process according to claim 26 wherein the fatty acid source is behenic acid glycerol ester and the fatty acid lower alkyl esters comprise behenic acid methyl ester; and wherein the color of the behenic acid methyl ester is about 0.5 or less Lovibond yellow.

28. A process for preparing fatty acid polyol polyester comprising the steps of:

- (a) preparing high purity fatty acid lower alkyl esters by
  - (1) reacting a fatty acid glycerol ester with a lower alkyl alcohol in the presence of a catalyst to produce a product mixture of fatty acid lower alkyl ester, fatty acid glycerol ester and glycerol;
  - (2) separating the product mixture into a glycerol-containing phase and a fatty acid lower alkyl ester-containing phase;

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cont.
- (3) water-washing the fatty acid lower alkyl ester-containing phase at an elevated temperature and elevated pressure to remove at least a portion of by-products from the fatty acid lower alkyl ester-containing phase;
  - (4) fractionally distilling the resulting water-washed fatty acid lower alkyl ester; and
  - (5) collecting at least one fraction of highly purified fatty acid lower alkyl ester; and

- (b) transesterifying the highly purified fatty acid lower alkyl ester of the collected fraction with a polyol to obtain a fatty acid polyol polyester; and

wherein the high purity fatty acid lower alkyl esters have an acid value of less than about 1.0.

29. A process according to claim 28 wherein the lower alkyl alcohol is methanol and the fatty acid glycerol ester is behenic acid glycerol ester.

30. A process according to claim 29 wherein the polyol polyester has a triglyceride level of less than about 0.5%, by weight.

31. A process for preparing a linked esterified alkoxyated polyol comprising the steps of:

- (a) converting a source of fatty acids to a product mixture comprising fatty acid lower alkyl esters and by-products;
- (b) water-washing the product mixture at an elevated temperature and an elevated pressure to remove at least a portion of the by-products from the product mixture;
- (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters having an acid value of no greater than about 1.0;
- (d) reacting a polyol with an epoxide to form an alkoxyated polyol;
- (e) reacting the alkoxyated polyol with a linking segment to form a linked alkoxyated polyol; and
- (f) transesterification of the linked alkoxyated polyol with the high purity fatty acid lower alkyl esters.

32. High purity fatty acid lower alkyl esters made by a process comprising the steps of:

- (a) converting a source of fatty acids to a product mixture comprising fatty acid lower alkyl esters and by-products;
- (b) water-washing the product mixture at an elevated temperature and an elevated pressure to remove at least a portion of the by-products from the product mixture; and
- (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters;

wherein at least a portion of the fatty acids have from about 20 to about 24 carbon atoms and further wherein the high purity fatty acid lower alkyl esters have an acid value of no greater than about 1.0.

33. High purity fatty acid lower alkyl ester according to claim 32, wherein the fatty acid lower alkyl esters comprise behenic methyl ester.

34. High purity fatty-acid lower alkyl esters according to claim 33, wherein the color of the behenic acid methyl ester is about 0.5 or less Lovibond yellow.

35. An esterified polyol made by a process comprising the steps of:

- (a) converting a source of fatty acids to a product mixture comprising fatty acid lower alkyl esters and by-products;
- (b) water-washing the product mixture at an elevated temperature and an elevated pressure to remove at least a portion of the by-products from the product mixture; and
- (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters; and
- (d) esterifying a polyol with the high purity fatty acid lower alkyl esters to obtain an esterified polyol;

wherein at least a portion of the fatty acids have from about 20 to about 24 carbon atoms and further wherein the high purity fatty acid lower alkyl esters have an acid value of no greater than about 1.0.

36. An esterified polyol according to claim 35, wherein the esterified polyol comprises a polyol fatty acid polyester and less than about 0.5%, by weight, triglyceride.

37. An alkyl ester product made by a process comprising the steps of:

- (a) converting a source of fatty acid to a product mixture comprising fatty acid lower alkyl esters and by-products;
- (b) water-washing the product mixture at an elevated temperature and elevated pressure to remove at least a portion of the by-products from the product mixture; and
- (c) fractionally distilling the water-washed product mixture to obtain high purity fatty acid lower alkyl esters; and
- (d) making an alkyl ester product with the high purity fatty acid lower alkyl esters;

wherein the step of fractionally distilling is selected from the group consisting of fractionally distilling in the absence of base at a temperature of from about 325°F to about 475°F and fractionally distilling in the presence of base at a temperature of from about 475°F to about 530°F, and wherein at least a portion of the fatty acids have at least about 16 carbon atoms and further wherein the high purity fatty acid lower alkyl esters have an acid value of no greater than about 1.0.


38. An alkyl ester product according to claim 37, wherein the alkyl ester product comprises an esterified polyol selected from the group consisting of esterified alkoxyated glycerols, esterified linked alkoxyated glycerols, sucrose fatty acid polyesters and mixtures thereof.

39. An alkyl ester product according to claim 37, wherein the alkyl ester product comprises a polyol fatty acid polyester and less than about 0.5%, by weight, triglyceride.

The support for these amendments is found in the claims as originally filed. These amendments are being entered to bring the claims into conformance with, *inter alia*, 37 CFR §1.75; no new matter is added.

Respectfully submitted for Applicants,

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